AD-A120 836 CHEMICAL ANALYSIS SUPPORT: LIMITED ANALYSIS OF BRISTOL RHODE ISLAND WELL.. (U) LITTLE (ARTHUR D) INC CAMBRIDGE MA K BELTIS ET AL. SEP 82 DRXTH-TE-CR-2135 1/1

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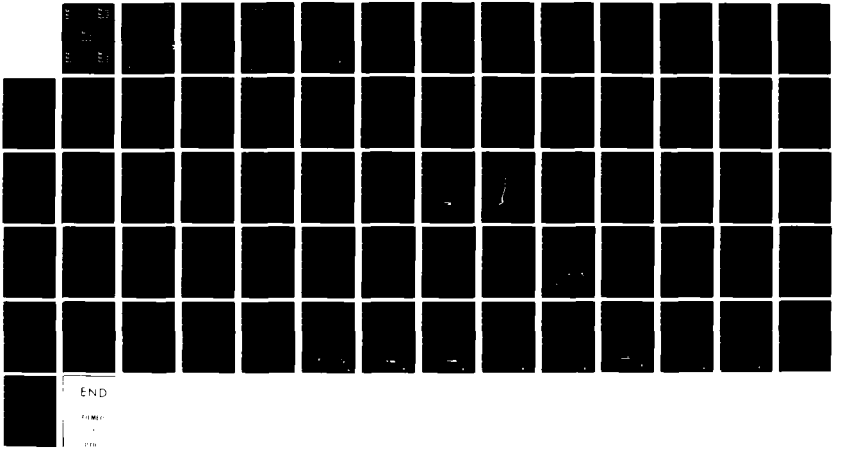

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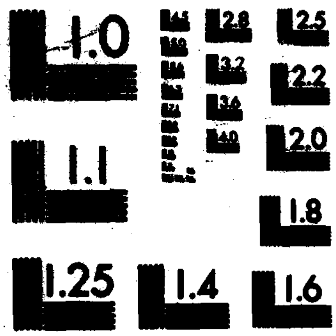
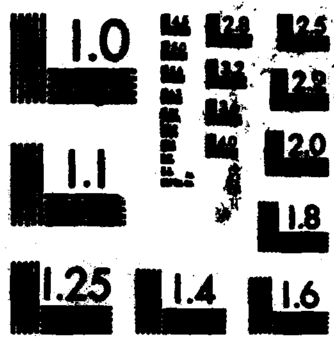
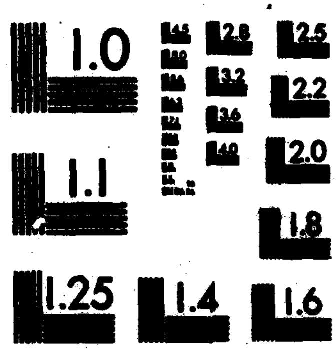




MICROCOPY RESOLUTION TEST CHART
NATIONAL BUREAU OF STANDARDS-1963-A



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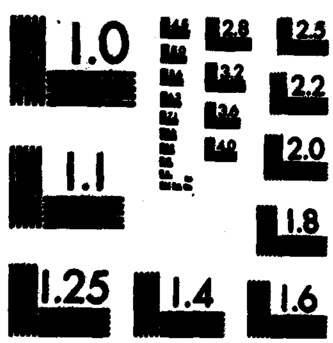

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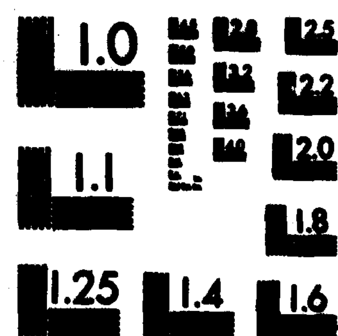

MICROCOPY RESOLUTION TEST CHART
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(12)

REPORT DRXTH-TE-CR-2135

CHEMICAL ANALYSIS SUPPORT: LIMITED ANALYSIS OF BRISTOL, RHODE ISLAND WELL WATER SAMPLES

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Christine Jones
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ARTHUR D. LITTLE, INC.
CAMBRIDGE, MA 02140

SEPTEMBER, 1982

FINAL TASK REPORT

DISTRIBUTION UNLIMITED: APPROVED FOR PUBLIC RELEASE

prepared for

U.S. Army Toxic and Hazardous Material Agency,
Aberdeen Proving Ground, Maryland 21010

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Volatile, Acid, Base/Neutral and Pesticide/PCB Priority Pollutants
1-1-dimethyl-hydrazine
Bristol, Rhode Island

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)
Well water samples collected from Bristol, Rhode Island were analyzed for volatile, acid, base/neutral and pesticide/PCB priority pollutants by the Federal Register Methods. Detection limits for the methods were determined following the May, 1980 USATHAMA QA program. In addition, attempts were made to detect the presence of UDMH in these field samples.

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TABLE OF CONTENTS

SUMMARY

Field samples collected for this task were analyzed for volatile, acid, base-neutral and pesticide priority pollutants using EPA methods. None of these pollutants were detected above the method detection limits determined from quality control samples.

In addition, experiments to develop an analytical method for 1,1-dimethyl-hydrazine were conducted but no satisfactory method was found in the time allotted. However, based on extraction and detection characteristics of similar compounds, it is estimated that 1 ppm of UDMH, if present would have been detected by gas chromatography/mass spectrometry analysis.

I. Sampling

On September 9, 1980 an employee of Arthur D. Little, Inc., met with USATHAMA personnel in Bristol, Rhode Island to assist in the collection of well water samples. Two distinct samples were collected, each consisting of two, 40-mL serum vials for the volatile organic analysis and two, gallon glass jars with teflon lined screw caps for the remaining analyses. Of the two samples, one represented the lower, aqueous phase present in the well, while the second sample represented the upper, oily phase present in the well. Table I-1 lists the samples collected and their subsequent laboratory sample numbers.

Table I-1

Samples Collected at Bristol Site

Field Sample Number	Description	Laboratory Number
BR 100 BR 102	2 gallons, aqueous phase	BR 100 (composite)
BR 101 BR 103	45-mL volatile organics, duplicate samples aqueous phase	BR 101 (BR 103)[1]
BR 104 BR 105	2 gallons, ~10% oil	BR 104 (composite)
BR 106 BR 107	45-mL volatile organics, duplicate samples oily phase	(BR 106)[1] BR 107

[1] Duplicate samples not analyzed

II. Sample Preparation/Analysis

Samples for the volatile organic analysis require no sample preparation and are analyzed directly by the purge and trap method[1] (Federal Register, Purgeables Method 624). One liter aliquots of BR 100 were extracted for acid and base-neutral analyses (Method 625) after initial pH adjustment, with three 60-mL volumes of methylene chloride. After drying each extract over sodium sulfate, it was concentrated to a final volume of 2-mL by Kuderna-Danish. Ten microliters of a d_{10}-anthracene standard solution was added as an internal standard just prior to analysis by gas chromatography/mass spectrometry[1] (Federal Register, Base-Neutrals Method 625).

A one-liter aliquot of BR 100 was also extracted for the priority pollutant pesticide analysis with three 60-mL volumes of 15 percent methylene chloride in hexane. The extract was concentrated to less than 10 mL and eluted through Florisil using 6 percent, 15 percent, and 50 percent diethylether in petroleum ether. After final concentration of each fraction to 10 mL, they were analyzed by gas chromatography using an electron capture detector[2]. In addition, 1 mL of BR 104, the oil phase, was diluted to 10 mL with hexane and analyzed by GC/ECD. No characteristic pesticide/PCB patterns were observed with this initial screen, making Florisil fractionation unnecessary.

Several attempts were made to analyze samples for unsymmetrical dimethylhydrazine (UDMH). An analytical standard (99%+ pure) was obtained from Aldrich Chemical Company (Cat. No. D16, 1608). Of the analytical methods cited in the literature, one was examined[3]. This method involves derivatization (with acetone), solvent extraction (pH adjustment with Base and CH_2Cl_2 extraction) and analysis by gas chromatography using a flame ionization detector. Laboratory samples were prepared by spiking neat UDMH into standard water (100 mg/L sulfate and chloride in distilled water) in the range of 0.1 to 10 parts per million. These spikes, along with aliquots of the two field samples, were analyzed as outlined in the method. No UDMH was detected from any of the samples analyzed. In addition to the OV-101 column recommended in the method, a 10% SP-2100 and a 5% SP-1000 column were examined. Column temperatures from 75°C to 200°C were tried in an effort to resolve the derivative, if present, from the solvent front. None of the conditions tried proved successful.

1,2-Diphenylhydrazine, one of the base-neutral priority pollutants, is routinely analyzed in our laboratories by GC/MS, with a calculated detection limit of 15 ppb (see Appendix C). Hydrazine can be reproducibly detected in the range of 30-50 ppb. Based on these data, it is estimated that $\leq$ 1 ppm UDMH would have been detected by GC/MS if it were present in the extracted sample.

In addition, the Bristol samples were examined based on mass spectral data. No evidence of UDMH was found in any of the three fractions examined (acid, base/neutral and volatiles).

III. Quality Control

The ability of Arthur D. Little, Inc. to perform priority pollutant analyses on samples for USATHAMA has been well documented in Tasks R902.35.03, R902.35.08, and R902.35.12. During the period of time the Bristol samples were being analyzed, Quality Control data for the priority pollutants were being generated under Task R902.35.08 of this program. Because of the Quick Response nature of the program and the limited number of samples that were to be analyzed for the Bristol site, authorization was given to reference this data for Quality Control purposes during sample analysis. Field samples obtained for Task R902.35.08 were spiked at five concentration levels and analyzed by the Federal Register methods. The detection limits were determined as stipulated in the May 1980 USATHAMA QA Program. These data are shown in the Appendices, including the slope (m), the correlation coefficient (R), and the detection limit at the 90% confidence level [x(d)], calculated by the method of Hubaux and Vos.

In addition, during analyses of the Bristol samples, samples were being analyzed concurrently for an EPA-sponsored contract. The quality control data from those EPA analyses are included in this report to demonstrate the fact that the laboratory and method were operating in control during analyses of the Bristol samples.

IV. RESULTS

The results of the analysis of the Bristol samples for volatile priority pollutants can be found in Appendix A. The total ion and reconstructed ion chromatograms of the Bristol sample show that only the internal standards are detected. These were added prior to sample analysis. The corresponding chromatograms for acid and base/neutral priority pollutant analyses reflect the character of the water sampled. The well from which the samples were obtained contained substantial quantities of an oil type material as described in the sampling section. The presence of this material provided a background spectra illustrated in the sample chromatograms for the acid and base/neutral fractions. The detection limits reported for the acid and base/neutral priority pollutants were generated under a separate ongoing task of this program as described in Section III. Because of the possibility of interferences from the hydrocarbons in the Bristol samples, the detection limits may be higher than the x(d) values reported for the quality control samples.

The results of the analysis of the florisil fractions of the Bristol samples indicate that no pesticide or PCB priority pollutants were detected at a level higher than the calculated detection limits.

V. References and Footnotes

[1] Federal Register 12/3/79. Vol. 44, No. 233. Purgeables Method 624, Base/Neutrals, Acids Method 625.

[2] U.S. EPA. Evaluation of Protocols for Pesticides and PCB's in Raw Wastewater. EPA-600/2-79-166.

[3] J. T. Veal. "The Analysis of Hydrazine, Monomethyl Hydrazine and 1,1-Dimethyl Hydrazine Using WCOT/GC Techniques." Air Force Syst. Command, Civ. Environ. Eng. Dev. Off., (Tech. Rep.) CEEDO-TR (U.S.), No. CEEDO-TR-78-14, Proc. Conf. Environ. Chem. Hydrazine Fuels, 1977, p 79-98 (1978).

APPENDIX A

Method for the Analysis of Organic Volatiles in Water

Priority Pollutant Volatile Organics Data

ANALYSIS OF ORGANIC VOLATILES

IN WATER

APPLICATION

METHOD USED TO DETERMINE THE CONCENTRATIONS OF THE FOLLOWING COMPOUNDS IN WATER SAMPLES;

CHLOROMETHANE
DICHLORODIFLUOROMETHANE
BROMOMETHANE
VINYL CHLORIDE
CHLOROETHANE
METHYLENE CHLORIDE
TRICHLOROFLUOROMETHANE
1,1-DICHLOROETHYLENE
1,1-DICHLOROETHANE
TRANS-1,2-DICHLOROETHYLENE
CHLOROFORM
1,2-DICHLOROETHANE
1,1,1-TRICHLOROETHANE
CARBON TETRACHLORIDE
BROMODICHLOROMETHANE
1,2-DICHLOROPROPANE
TRANS-1,3-DICHLOROPROPYLENE
TRICHLOROETHYLENE
BENZENE
CIS-1,3-DICHLOROPROPYLENE
DIBROMOCHLOROMETHANE
1,1,2-TRICHLOROETHANE
BROMOFORM
1,1,2,2-TETRACHLOROETHANE

1,1,2,2-TETRACHLOROETHYLENE

TOLUENE

CHLOROBENZENE

ETHYL BENZENE

A. TESTED CONCENTRATION RANGE:
5-50 UGL FOR EACH

B. SENSITIVITY; SEE FEDERAL REGISTER VOL. 44, NO. 233.

C. DETECTION LIMIT:

CHLOROMETHANE	18 UGL
DICHLORODIFLUOROMETHANE	42 UGL
BROMOMETHANE	15 UGL
VINYL CHLORIDE	21 UGL
CHLOROETHANE	28 UGL
METHYLENE CHLORIDE	18 UGL
TRICHLOROFLUOROMETHANE	14 UGL
1,1-DICHLOROETHYLENE	18 UGL
1,1-DICHLOROETHANE	13 UGL
TRANS-1,2-DICHLOROETHYLENE	14 UGL
CHLOROFORM	10 UGL
1,2-DICHLOROETHANE	10 UGL
1,1,1-TRICHLOROETHANE	17 UGL
CARBON TETRACHLORIDE	15 UGL
BROMODICHLOROMETHANE	11 UGL
1,2-DICHLOROPROPANE	11 UGL
TRANS-1,3-DICHLOROPROPYLENE	10 UGL
TRICHLOROETHYLENE	13 UGL
BENZENE	12 UGL
CIS-1,3-DICHLOROPROPYLENE	8 UGL
DIBROMOCHLOROMETHANE	9 UGL
1,1,2-TRICHLOROETHANE	11 UGL
BROMOFORM	9 UGL
1,1,2,2-TETRACHLOROETHANE	9 UGL
1,1,2,2-TETRACHLOROETHYLENE	11 UGL

TOLUENE	12 UGL
CHLOROBENZENE	12 UGL
ETHYL BENZENE	12 UGL

D. INTERFERENCES; SEE FEDERAL REGISTER 12/3/79 VOL. 44, NO. 233

E. ANALYSIS RATE; AFTER TUNING OF INSTRUMENT, ONE ANALYST CAN ANALYZE 6-7 FIELD SAMPLES IN AN 8-HOUR DAY

CHEMISTRY

CH3C1 CHLOROMETHANE
CAS RN 74-87-3
MELTING PT - 97C BOILING PT - 23.7C

CCL2F2 DICHLORODIFLUOROMETHANE
CAS RN 75-71-8
MELTING PT - 158C BOILING PT - 29.8C

CH3BR BROMOMETHANE
CAS RN 74-83-9
MELTING PT - 93.66C BOILING PT 3.56C

C2H3CL VINYL CHLORIDE
CAS RN 9003-22-9
MELTING PT - 160C BOILING PT - 14C

C2H5CL CHLOROETHANE
CAS RN 75-00-3
MELTING PT - 138.7C BOILING PT 12.3C

CH2CL2 METHYLENE CHLORIDE
CAS RN 75-09-2
MELTING PT - 97C BOILING POINT 39.8-40C

CCL3F TRICHLOROFLUOROMETHANE
CAS RN 75-69-4
MELTING PT - 111C BOILING PT 23.7C

C2H2CL2 1,1-DICHLOROETHYLENE
CAS RN 75-35-4
MELTING PT - 122C BOILING PT 30-32C

C2H4CL2 1,1-DICHLOROETHANE
CAS RN 75-34-3
MELTING PT - 97C BOILING PT 57C

C2H2CL2 TRANS-1,2-DICHLOROETHYLENE
CAS RN 156-60-5
MELTING PT - 50C BOILING PT 48C

CHCL3 CHLOROFORM
CAS RN 865-49-6
MELTING PT - 63.5C BOILING PT 60.3-61.5C

C2H4CL2 1,2-DICHLOROETHANE
CAS RN 107-06-2
MELTING PT - 35C BOILING PT 83C

C2H3CL3 1,1,1-TRICHLOROETHANE
CAS RN 71-55-6
MELTING PT - 35C BOILING PT 74-76C

CCL4 CARBON TETRACHLORIDE
CAS RN 56-23-5
MELTING PT - 23C BOILING PT 77C

CHBRCL2 BROMODICHLOROMETHANE
CAS RN 75-27-4
MELTING PT - 55C BOILING PT 87C

C3H6CL2 1,2-DICHLOROPROPANE
CAS RN 78-87-5
MELTING PT - 100C BOILING PT 95-96C

C3H4CL2 TRANS-1,3-DICHLOROPROPYLENE
CAS RN 78-88-6
BOILING PT 112C

C2HCL3 TRICHLOROETHYLENE
CAS RN 79-01-6
MELTING PT - 87C BOILING PT 87C

C6H6 BENZENE
CAS RN 71-43-2
MELTING PT 5.5C BOILING PT 80.2C

C3H4CL2 CIS-1,3-DICHLOROPROPYLENE
CAS RN 78-88-6
BOILING PT 104.3C

CHBR2CL DIBROMOCHLOROMETHANE
CAS RN 124-48-1
MELTING PT - 22C BOILING PT 119-120C

C2H3CL3 1,1,2-TRICHLOROETHANE
CAS RN 79-00-5
MELTING PT - 37C BOILING PT 110-115C

CHBR3 BROMOFORM
CAS RN 75-25-2
MELTING PT 8C BOILING PT 146-150C

C2H2CL4 1,1,2,2-TETRACHLOROETHANE
CAS RN 79-34-5
MELTING PT - 43C BOILING PT 147C

C2CL4 1,1,2,2-TETRACHLOROETHYLENE
CAS RN 127-18-4
MELTING PT - 22C BOILING PT 121C

C_7H_8 TOLUENE
CAS RN 108-88-3
MELTING PT - 93C BOILING PT 111C

C6H5CL CHLOROBENZENE
CAS RN 108-90-7
MELTING PT - 46C BOILING PT 132C

C8H10 ETHYL BENZENE
CAS RN 100-41-4
MELTING PT - 95C BOILING PT 136C
USE CAUTION IN HANDLING. POTENTIAL TOXIC INHALATION, AND SKIN ABSORPTION HAZARDS EXIST.

APPARATUS

A. INSTRUMENTATION;
PURGE AND TRAP - TEKMAR MODEL LSC-1 LIQUID SAMPLE CONCENTRATOR

GAS CHROMATOGRAPH - FINNIGAN 4000 GAS CHROMATOGRAPH/MASS SPECTROMETER WITH A 6110 DATA SYSTEM

B. PARAMETERS;
AMBIENT TEMPERATURE TRAP - 8" OF STAINLESS STEEL TUBING PACKED WITH 0.3" 3% OV-1: 5" TENAX GC; 2.7" SILICA GEL AND GLASS WOOL.

COLUMN - 0.2% CARBOWAX 1500 ON 80/100 CARBOPACK PACKED IN 6-FT X 2 MM ID GLASS COLUMN

GAS FLOW - HELIUM AT 40 ML/MIN

TEMPERATURE -
TRAP - 180C
COLUMN - INITIAL 60C FOR 3 MIN PROGRAM TO 165C AT 8C/MIN. HOLD AT 165C FOR 15 MIN.

INJECTION VOLUME - 5 ML PURGED

DETECTOR - MASS SPECTROMETER: ELECTRON IMPACT DATA ACQUIRED OVER MASS RANGE 21-260 AMU, SCANNING EVERY 2 SECONDS

RETENTION TIME - SEE FEDERAL REGISTER 12/3/79 Vol. 44, NO. 233.

C. HARDWARE/GLASSWARE; SEE FEDERAL REGISTER 12/3/79 VOL. 44, NO. 233.

D. CHEMICALS; SEE FEDERAL REGISTER 12/3/79 VOL. 44, NO. 233.

SUPELCO INC. PURGABLE A, CATALOG #4-8815, 0.2 MG/ML CONCENTRATION

SUPELCO INC. PURGABLE B, CATALOG #4-8816, 0.2 MG/ML CONCENTRATION

SUPELCO INC. PURGABLE C, CATALOG #4-8817, 0.2 MG/ML CONCENTRATION

SUPELCO INC. INTERNAL STANDARD, CATALOG #4-8823, 20 MG/ML CONCENTRATION

STANDARDS

A. CALIBRATION STANDARDS

TALK 1 ML EACH OF SUPELCO, INC. PURGEABLES A, B AND C (SEE SECTION APPARATUS D) AND 1 ML OF METHANOL TO MAKE MASTER SPIKE SOLUTION AT 50 UG/ML CONCENTRATIONS. PREPARE WORKING CALIBRATION STANDARDS USING MILLI-Q WATER AS FOLLOWS.

CAL	ML STOCK/2 ML	FINAL CONC.
CAL 1	2 ML	50 UG/ML
CAL 2	1 ML	25 UG/ML
CAL 3	.5 ML	12.5 UG/ML
CAL 4	.2 ML	5 UG/ML

NEW STANDARDS ARE PREPARED FOR EVERY VOA SET WHICH IS ANALYZED OR AT A MINIMUM EVERY TWO WEEKS. THE STANDARDS ARE STORED IN SEALED VIALS WHICH ARE PACKED IN PAINT CANS FILLED WITH ACTIVATED CHARCOAL AND REFRIGERATED. IN ADDITION, SPIKE ALL CALIBRATION STANDARDS AND FIELD SAMPLES WITH SUPELCO INTERNAL STANDARD STOCK AS FOLLOWS:

INTERNAL STANDARD STOCK = 20 MG/ML BROMOCHLOROMETHANE

20 MG/ML 1,4-DICHLOROBUTANE

20 MG/ML 1-CHLORO-2-BROMO PROPANE

TAKE 10 UL OF INTERNAL STANDARD STOCK AND DILUTE TO 10 MLS WITH METHANOL TO MAKE A MASTER INTERNAL STANDARD SPIKING STOCK AT 20 MG/L.

SPIKE 5 ML SAMPLES WITH 5 UL MASTER INTERNAL STANDARD SPIKING STOCK TO GIVE FINAL CONCENTRATION OF 20 UGL.

SPIKE 2 ML CALIBRATION STANDARDS WITH 2 UL MASTER INTERNAL STANDARD SPIKING STOCK TO GIVE FINAL CONCENTRATION OF 20 UGL.

B. CONTROL SPIKES;
PREPARE THE SAME WAY AS THE CALIBRATION STANDARDS ONLY PREPARE IN 2 ML NATURAL WATER

PROCEDURE

SEE FEDERAL REGISTER 12/3/79 VOL. 44, NO. 233.

CALCULATIONS

USING CONTROL SPIKE SAMPLE CONCENTRATION AS CALCULATED FROM DAILY CALIBRATION, PLOT UGL ADDED VERSUS UGL FOUND, BY THE METHOD OF HUBAUX AND VOS, USING THE DETECTION LIMIT TAPE SUPPLIED BY USATHAMA. CORRECT ALL FIELD SAMPLE CONCENTRATIONS USING THE SLOPE OF THE LINEAR REGRESSION LINE.

TABLE A-1

QC NATURAL WATER SPIKES[a]
Task R 902.35.08
PRIORITY POLLUTANT CHEMICAL ANALYSIS (µg/L)
PURGEABLE (VOLATILES)

SPIKE LEVEL		+5	+12.5	+20	+25	+50	R	X_d
Chloromethane		--	4.7	NA	19.2	36.9	.991	18.1
Dichlorodifluoromethane		3.8	12.5	NA	23.2	33.7	.974	42.3
Bromomethane		6.9	12.5	NA	22.9	37.5	.996	14.7
Vinyl chloride		7.4	14.4	NA	23.6	36.9	.993	20.6
Chloroethane		6.9	12.0	NA	26.3	39.9	.986	28.4
Methylene chloride		3.7	7.9	10.3	15.3	23.6	.988	17.6
Trichlorofluoromethane		6.4	13.9	19.1	25.7	41.1	.993	13.6
1,1-Dichloroethylene		6.4	13.6	NA	25.3	41.2	.983	18.4
1,1-Dichloroethane		5.7	14.5	18.2	28.6	50.8	.993	12.9
Trans-1,2-dichloroethylene		5.3	13.7	16.8	26.5	46.8	.993	13.5
Chloroform		4.5	12.7	17.1	25.2	45.6	.996	9.9
1,2-Dichloroethane		5.3	14.9	18.8	26.7	49.2	.996	9.7
1,1,1-Trichloroethane		5.6	12.8	14.7	24.8	42.4	.988	17.2
Carbon tetrachloride		5.6	12.2	NA	23.4	39.5	.996	15.0
Bromodichloromethane		5.0	11.0	NA	22.2	39.1	.998	11.4
1,2-Dichloropropane		6.5	14.7	18.6	26.8	46.9	.996	10.6
Trans-1,3-dichloropropylene		5.2	13.0	17.2	25.6	48.1	.996	9.5
Trichloroethylene		5.2	12.4	15.2	23.6	42.4	.994	12.6
Benzene		4.4	13.3	17.2	27.3	49.3	.994	12.5
Cis-1,3-dichloropropylene		5.0	12.1	NA	24.5	45.3	.999	7.6
Dibromochloromethane		4.5	10.2	NA	21.3	38.8	.999	9.1
1,1,2-Trichloroethane		4.7	13.3	NA	25.0	45.4	.998	11.4
Bromoform		4.2	8.6	13.1	18.5	38.9	.997	9.1
1,1,2,2-Tetrachloroethane		4.8	13.6	18.1	26.6	49.8	.997	9.1
1,1,2,2-Tetrachloroethylene		5.0	12.0	NA	23.2	41.4	.998	10.6
Toluene		5.1	13.2	17.3	27.2	50.1	.995	11.6
Chlorobenzene		5.5	13.2	16.7	26.3	47.5	.994	12.5
Ethyl benzene		5.3	13.4	17.1	27.2	49.8	.994	12.4

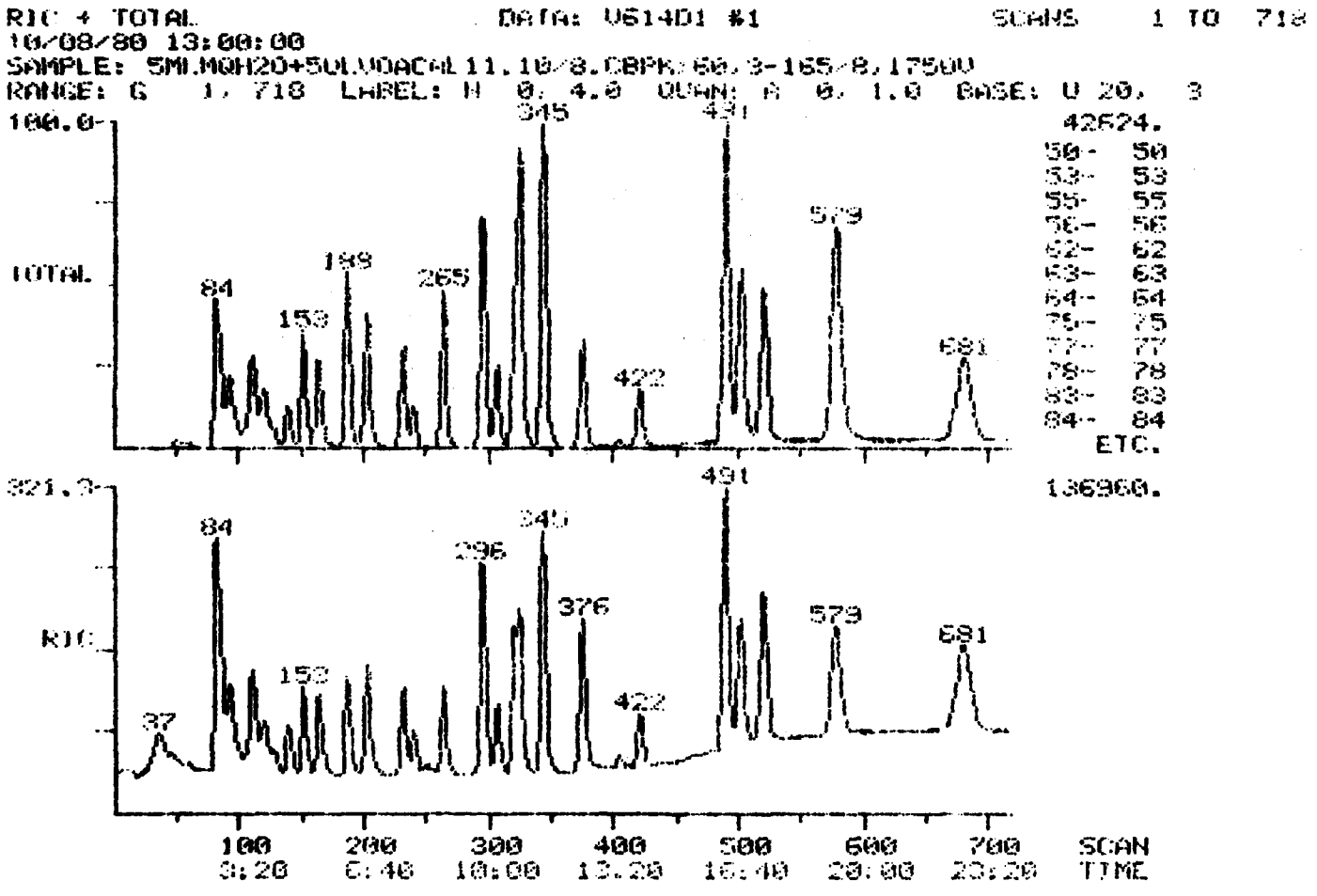
NA = Not Analyzed
[a]Field sample from uncontaminated background well bordering a U.S. Army installation site.

TABLE A-2

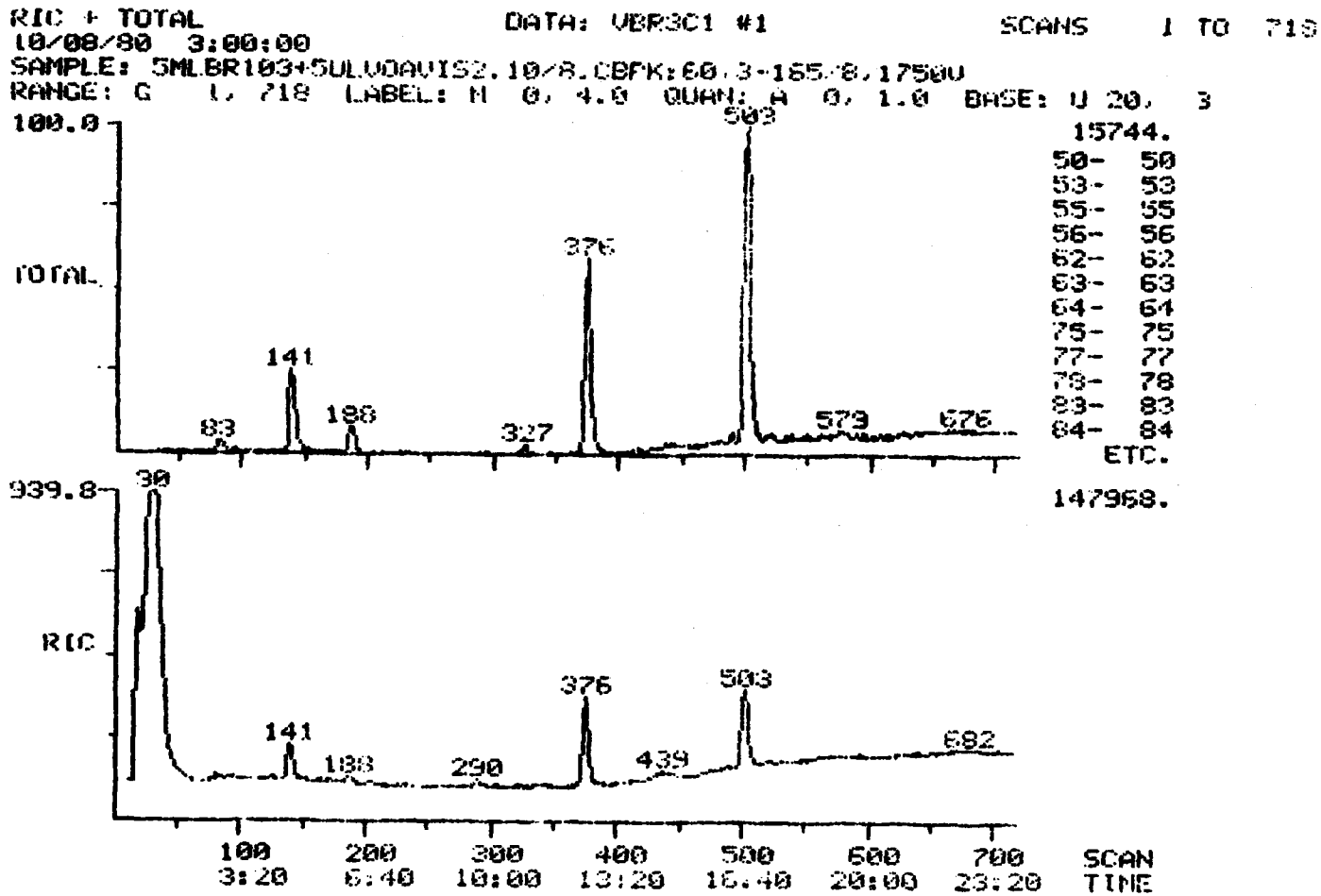
EPA QUALITY CONTROL DATA SUMMARY
PURGEABLE (VOLATILES)

	Method Reference Standard[a]					Raw Wastewater Spike[b]		
	$\bar{P}$	Sp	% Sp			$\bar{P}$	Sp	% Sp
Chloromethane	104[c]	20	19			-		
Dichlorodifluoromethane	-					-		
Bromomethane	93	31	34			114	/	6
Vinyl chloride	72[b]	4[b]	64			122	97	80
Chloroethane	102	36	35			121	13	11
Methylene chloride	105	18	17			116	5	4
Acrolein	114[b]	17	15			134	15	12
Trichlorofluoromethane	71	46	65			118	8	/
Acrylonitrile	110	14	13			124	19	16
1,1–Dichloroethylene	90	22	25			108	9	8
1,1–Dichloroethane	111	20	18			117	4	3
Trans–1,2–dichloroethylene	104	13	13			105	6	6
Chloroform	107	20	19			122	16	13
1,2–Dichloroethane	104	11	11			102	9	9
1,1,1–Trichloroethane	105	50	19			110	17	16
Carbon tetrachloride	94	22	24			98	21	21
Bromodichloromethane	102	10	10			96	16	17
1,2–Dichloropropane	102	7	7			90	8	8
Trans–1,3–dichloropropylene	105	14	13			102	2	2
Trichloroethylene	104	13	12			102	21	20
Benzene	104	6	6			110	8	7
Cis–1,3–dichloropropylene	101	19	19			93	4	4
Dibromochloromethane	106	7	/			92	16	18
1,1,2–Trichloroethane	110	9	8			107	15	14
Bromoform	104	17	16			83	20	24
1,1,2,2–Tetrachloroethane	105	11	11			108	10	9
1,1,2,2–Tetrachloroethylene	98	30	30			94	24	26
Toluene	103	15	15			102	16	16
Chlorobenzene	104	16	15			102	16	16
Ethyl benzene	96	22	23			98	20	20

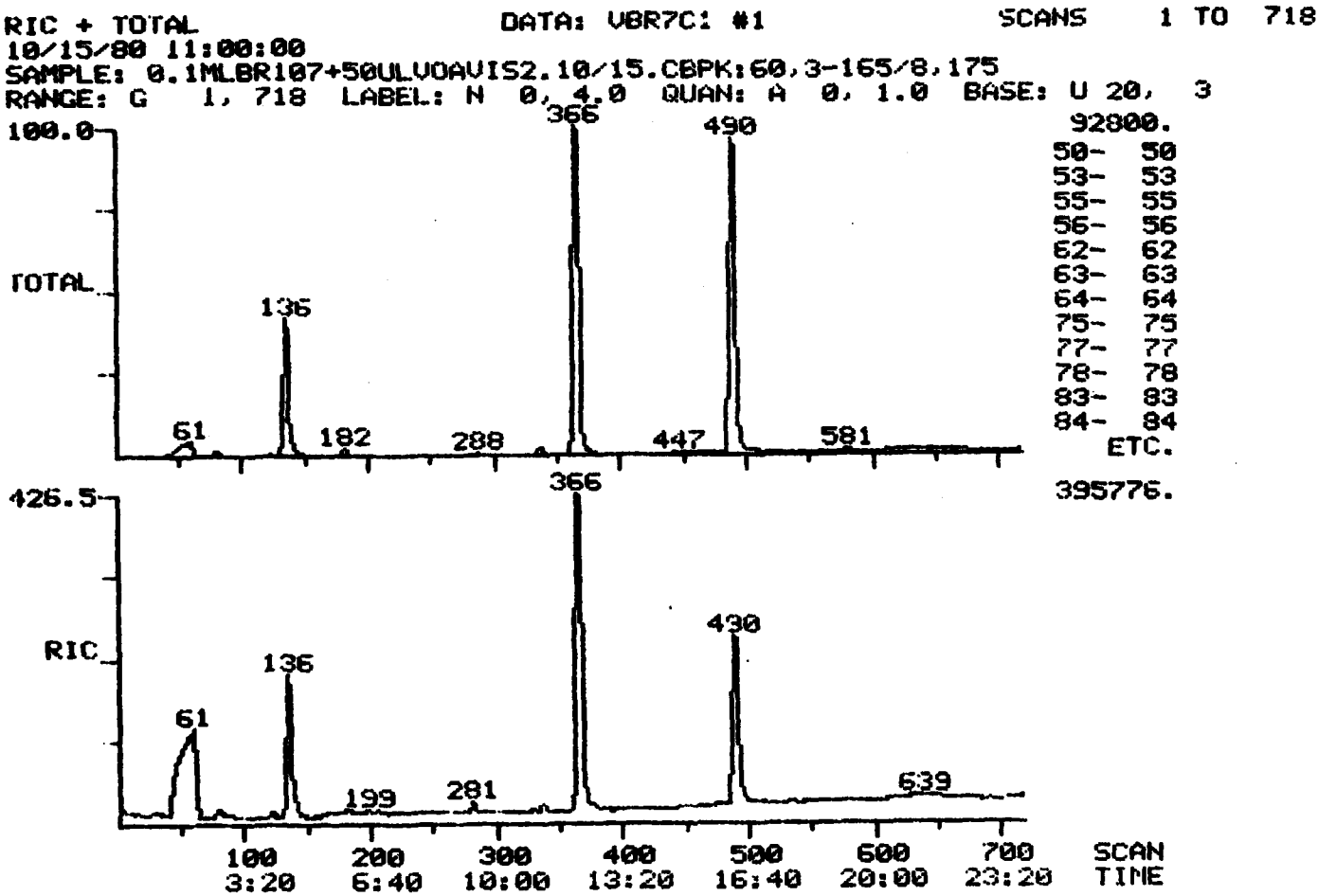
(a) Based on 5 data points, spiked concentrations = 10µg/L. and 25µg/L.
(b) Based on 4 data points, spiked concentrations = 10µg/L. and 25µg/L.
(c) Based on 2 data points, spiked concentrations = 10µg/L. and 25µg/L.
See text page 5, for explanation of Statistics.



Volatile Organics Calibration Mix



BR 103 Volatile Fraction



BR 107 Volatile Fraction

APPENDIX B

Method for the Analyses of Priority Pollutant Acids in Water

Priority Pollutant Acids Data

ANALYSIS OF ACID PRIORITY POLLUTANTS IN WATER

APPLICATION:

METHOD USED TO DETERMINE CONCENTRATIONS OF THE FOLLOWING COMPOUNDS IN WATER SAMPLES

- 2-CHLOROPHENOL
- 2-NITROPHENOL
- PHENOL
- 2,4-DIMETHYLPHENOL
- 2,4-DICHLOROPHENOL
- 2,4,6-TRICHLOROPHENOL
- 4-CHLORO-3-CRESOL
- 2,4-DINITROPHENOL
- 4,6-DINITRO-2-CRESOL
- PENTACHLOROPHENOL
- 4-NITROPHENOL

A. TESTED CONCENTRATION RANGE; 5 to 100 UGL FOR EACH

B. SENSITIVITY; SEE FEDERAL REGISTER 12/3/79 VOL. 4, NO. 233

C. DETECTION LIMIT;

2-CHLOROPHENOL	33 UGL
2-NITROPHENOL	25 UGL
PHENOL	19 UGL
2,4-DIMETHYLPHENOL	48 UGL
2,4-DICHLOROPHENOL	26 UGL
2,4,6-TRICHLOROPHENOL	22 UGL
4-CHLORO-3-CRESOL	22 UGL
2,4-DINITROPHENOL	73 UGL
4,6-DINITRO-2-CRESOL	45 UGL
PENTACHLOROPHENOL	16 UGL
4-NITROPHENOL	58 UGL

D. INTERFERENCES; SEE FEDERAL REGISTER 12/3/79 VOL 4, NO. 233

E. ANALYSIS RATE; AFTER TUNING OF INSTRUMENT, ONE ANALYST CAN ANALYZE 8 SAMPLES IN A 8-HOUR DAY.

CHEMISTRY;

C6H5CLO 2-CHLOROPHENOL
CAS RN 95-57-8
MELTING PT 8C BOILING PT 175-176C

C6H5NO3 2-NITROPHENOL
CAS RN 88-75-5
MELTING PT 44-45C BOILING PT 214-216C

C6H5OH PHENOL
CAS RN 108-95-2
MELTING PT 39.5-41.5C BOILING PT 181C

C8H10O 2,4-DIMETHYLPHENOL
CAS RN 105-67-9
METLING PT 25.4-26.0C BOILING PT 212C

C6H4CL2O 2,4-DICHLOROPHENOL
CAS RN 120-83-2
MELTING PT. 42-43C BOILING PT 209-210C

C6H3CL3O 2,4,6-TRICHLOROPHENOL
CAS RN 88-06-2
MELTING PT 64-66C BOILING PT 246C

C7H7CLO 4-CHLORO-3-CRESOL
CAS RN 59-50-7
MELTING PT 65-68C BOILING PT 235C

C6H4N2O5 2,4-DINITROPHENOL
CAS RN 51-28-5
METLING PT 106-108C

C7H6N2O5 4,6-DINITRO-2-CRESOL
CAS RN 534-52-1
MELTING PT 87.5C

C6CL5OH PENTACHLOROPHENOL
CAS RN 87-86-5
METLING PT 188-191C BOILING PT 309-310C

C6H5NO3 4-NITROPHENOL
CAS RN 100-02-7
MELTING PT 112-114C BOILING PT 279C

USE CAUTION IN HANDLING. POTENTIAL TOXIC INHALATION, AND SKIN ABSORPTION HAZARDS EXIST.

APPARATUS;

A. INSTRUMENTATION;
GAS CHROMATOGRAPH - FINNIGAN 4000 GAS CHROMATOGRAPH/MASS SPECTROMETER WITH A 6110 DATA SYSTEM

B. PARAMETERS;
COLUMN-1% SP 2250 on 100/120 SUPELCOPORT,
6 FT X 2 MM ID GLASS COLUMN
GAS FLOW - HELIUM GAS AT 30 ML/MIN
TEMPERATURE - HELD AT 50C FOR 4 MINUTES
PROGRAMMED AT 265C AT 8C/MINUTE
HELD CONSTANT AT 265C FOR 30
MINUTES
INJECTOR VOLUME - 2 UL
DETECTOR - MASS SPECTROMETER SET TO ACQUIRE
ELECTRON IMPACT DATA OVER THE MASS
RANGE 41-425 AMV.
SCANNING EVERY 3 SECONDS
RETENTION TIMES - SEE FEDERAL REGISTER
12/3/79 VOL. 4, NO. 233

C. HARDWARE/GLASSWARE; SEE FEDERAL REGISTER
12/3/79 VOL. 4, NO. 233

D. CHEMICALS; SEE FEDERAL REGISTER 12/3/79
VOL. 4, NO. 233

STANDARDS;

A. CALIBRATION STANDARDS; PREPARE STOCK SOLUTION BY MIXING 12.5 MG OF EACH OF THE FOLLOWING ACIDS AND DILUTING TO 25 ML IN METHANOL:

2-CHLOROPEHNOL	4-CHLORO-3-CRESOL
2-NITROPHENOL	2,4-DINITROPHENOL
PHENOL	4,6-DINITRO-2-CRESOL
2,4-DIMETHYLPEHNOL	PENTACHLOROPHENOL
2,4-DICHLOROPHENOL	4-NITROPHENOL
2,4,6-TRICHLOROPHENOL	

FINAL CONCENTRATION IS 0.5 MG/ML
PREPARE WORKING CALIBRATION STANDARDS USING METHYLENE CHLORIDE AS FOLLOWS:

CAL	UL STOCK/2ML CH2CL2	FINAL CONC.
A	20 UL	5 UG/ML
B	40 UL	10 UG/ML
C	100 UL	25 UG/ML
D	200 UL	50 UG/ML
E	300 UL	75 UG/ML

10 UL INTERNAL STANDARD SPIKING SOLUTION AND 10 UL D10 ANTHRANCENE WERE ADDED TO ALL CALIBRATION STANDARDS AND SAMPLES.

THE INTERNAL STANDARD SPIKING SOLUTION WAS PREPARED AS FOLLOWS;

53.3 MG OCTAFLUOROBIPHENYL AND 51.45 MG 2,4-DIBROMOPHENOL WERE ADDED TO 50 ML VOLUMETRIC FLASK AND DILUTED TO MARK IN METHANOL

FINAL CONCENTRATIONS

1.066 MG/ML OCTAFLUOROBIPHENYL
1.029 MG/ML 2,4-DIBROMOPHENOL

D10 ANTHRACENE STOCK WAS PREPARED AS FOLLOWS:
DILUTE 50 MG D10 ANTHRACENE TO 50 ML IN METHYLENE CHLORIDE. FINAL CONCENTRATION IS 1.0 MG/ML

B. CONTROL SPIKES: A LITER OF NATURAL WATER WAS SPIKED WITH THE ACID STOCK SOLUTION (0.5 MG/ML) AS FOLLOWS:

QC	UL STOCK/L	FINAL CONC.
1	BLANK	-
2	10 UL	5 UG/L
3	20 UL	10 UG/L
4	40 UL	20 UG/L
5	100 UL	50 UG/L
6	200 UL	100 UG/L

IN ADDITION, QUALITY CONTROL SAMPLES (1L) WERE SPIKED WITH 10 UL INTERNAL STANDARD SOLUTION. PRIOR TO ANALYSIS ALL 2ML EXTRACTS WERE SPIKED WITH 10 UL D10 ANTHRACENE STOCK

PROCEDURE

SEE FEDERAL REGISTER 12/3/79 VOL. 44, NO. 233

CALCULATIONS:

CALCULATE UGL FOR EACH SAMPLE FROM DAILY CALIBRATION CURVE. USING CALCULATED CONTROL SPIKE CONCENTRATIONS, PLOT UGL ADDED VERSUS UGL FOUND USING DETECTION LIMIT TAPE SUPPLIED BY USATHAMA. CORRECT ALL FIELD SAMPLE CONCENTRATIONS USING THE SLOPE OF THE LINEAR REGRESSION LINE.

REFERENCES:

FEDERAL REGISTER 12/3/79 VOL. 44, NO. 233.
PAGES 69540-69547

TABLE B-1

QC NATURAL WATER SPIKES[a]

Task R902.35.08

PRIORITY POLLUTANT CHEMICAL ANALYSIS (µg/L)
ACIDS

SPIKE LEVEL	-	+5	+10	+20	+50	+100	R	x(d)
2-Chlorophenol	ND	4.8	7.9	19.9	54.4	87.0	.991	33
2-Nitrophenol	ND	3.4	5.3	15.4	46.4	79.8	.995	25
Phenol	ND	3.0	5.5	13.5	38.4	68.0	.997	19
2,4-Dimethylphenol	ND	1.9	5.1	16.1	42.6	63.4	.982	48
2,4-Dichlorophenol	ND	4.4	7.0	18.3	52.0	87.8	.994	26
2,4,6-Trichlorophenol	ND	4.2	6.1	16.8	49.2	86.0	.996	22
4-Chloro-3-cresol	ND	3.8	5.6	15.6	45.2	78.8	.996	22
2,4-Dinitrophenol	ND	-	0.1	1.1	10.5	54.6	.961	73
4,6-Dinitro-2-cresol	ND	0.2	0.4	2.6	16.9	57.0	.983	45
Pentachlorophenol	ND	3.0	5.0	13.0	35.0	81.0	.998	16
4-Nitrophenol	ND	2.2	1.8	5.5	13.2	48.6	.974	58

[a]Field sample from uncontaminated background well bordering a U.S. Army installation site.

ND = none detected

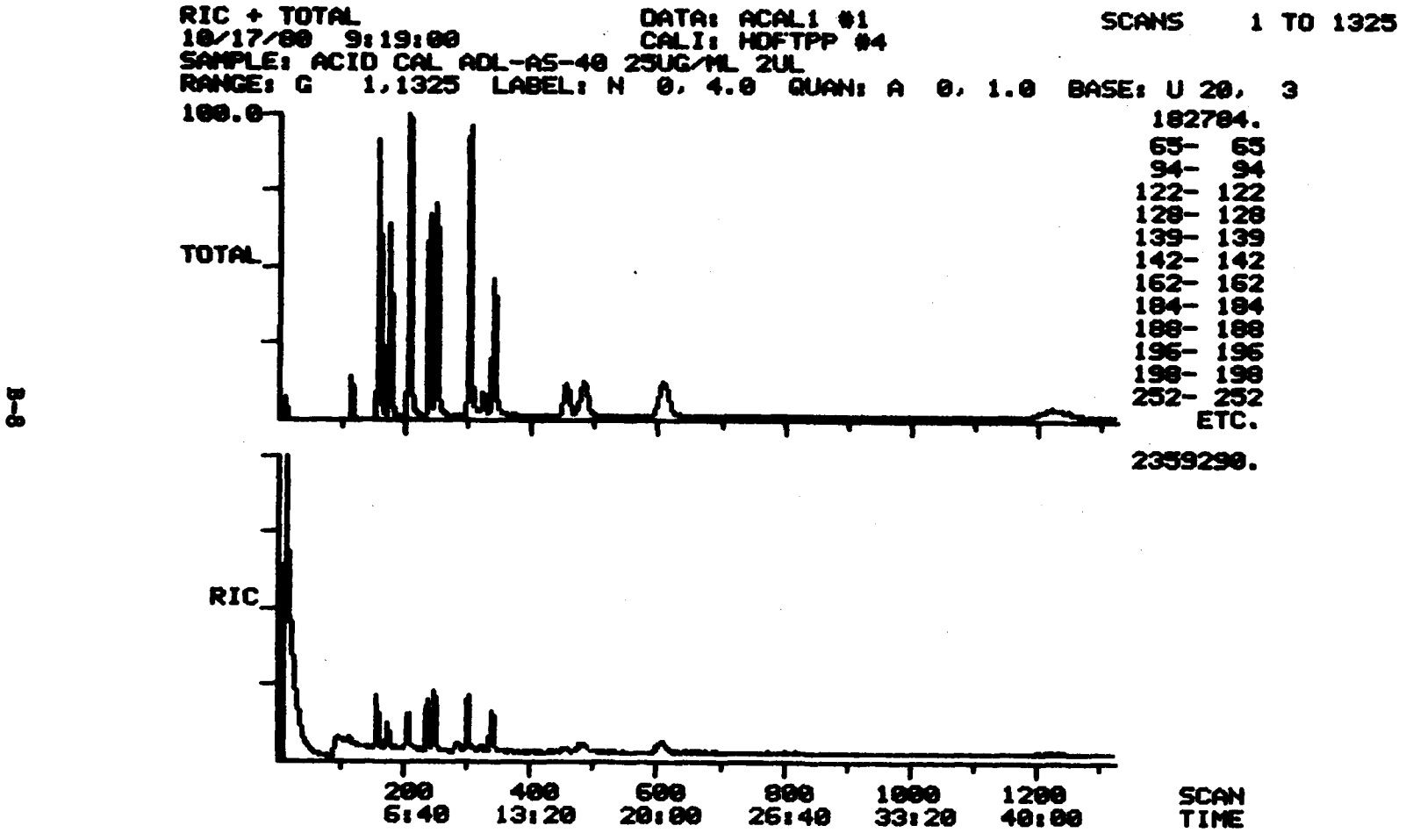
TABLE B-2

EPA QUALITY CONTROL DATA SUMMARY
ACIDS

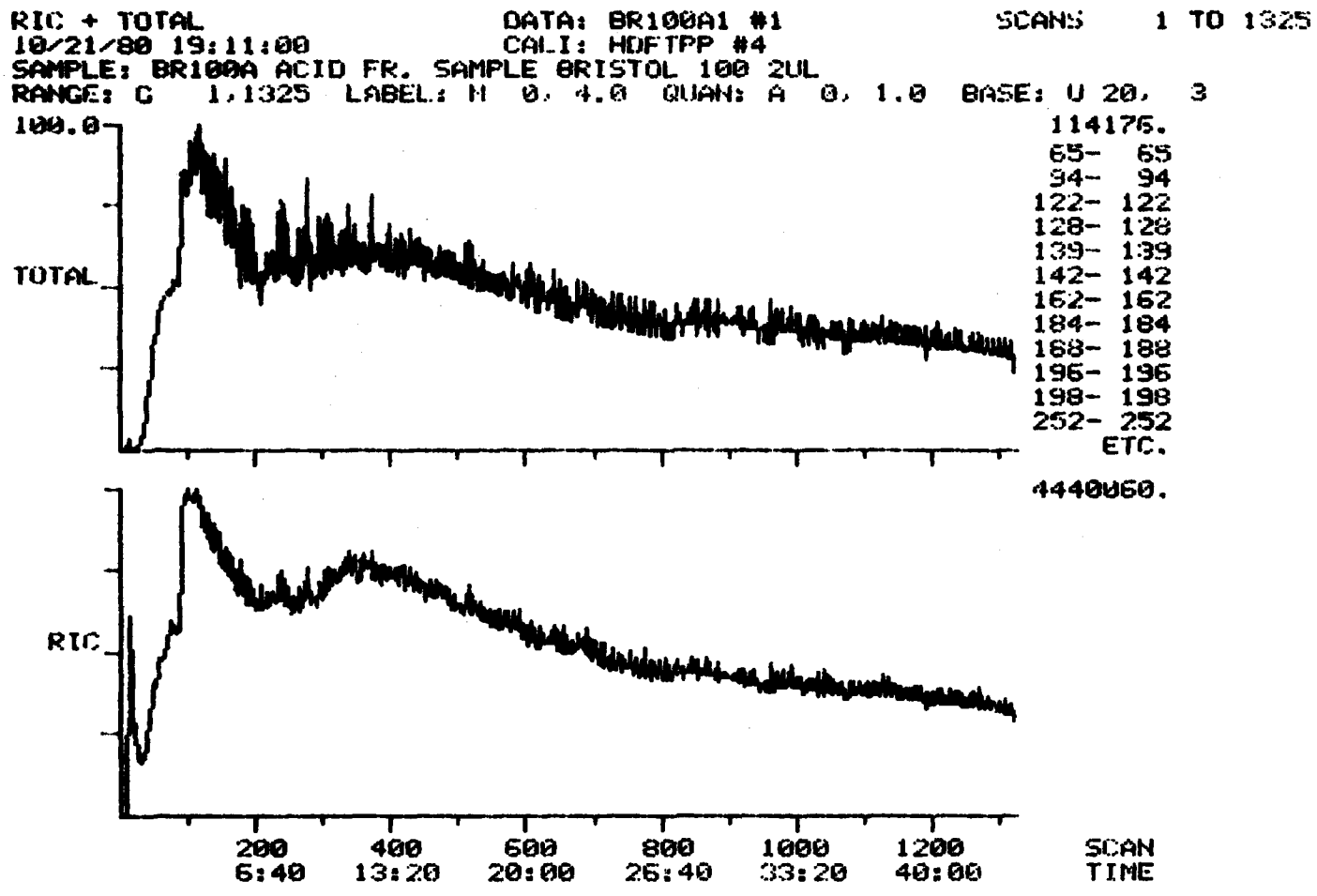
	Method Reference Standard[a]					Raw Wastewater Spike[a]		
	$\bar{P}$	Sp	% Sp			$\bar{P}$	Sp	% Sp
2-Chlorophenol	84	13	16			97[b]	9	10
2-Nitrophenol	84	7	9			99[b]	2	2
Phenol	42	11	25			61	24	40
2,4-Dimethylphenol	78	11	14			71[b]	31	44
2,4-Dichlorophenol	89	12	13			96	18	19
2,4,6-Trichlorophenol	98	12	12			108[b]	14	13
4-Chloro-3-cresol	86	10	11			82	39	48
2,4-Dinitrophenol	69	9	14			79	2	3
4,6-Dinitro-2-cresol	84	11	13			74	17	22
Pentachlorophenol	79	13	17			64	29	45
4-Nitrophenol	33	6	18			32	3	10

(a) Based on 4 data points, spiked concentration = 50 µg/L
The raw waste water spikes were ground water samples associated with a municipal land fill. The data was generated for an EPA sponsored program during the same time period the Bristol samples were analyzed

(b) Based on 3 data points, spiked concentration = 50 µg/L
See text, page 5, for explanation of statistics



Acid Calibration Mix



BR 100 Acid Fraction

APPENDIX C

Method for the Analysis of Priority Pollutant Base/ Neutrals on Water

Priority Pollutant Base/Neutral Data

ANALYSIS OF BASE/NEUTRALS IN WATER

APPLICATION

METHOD USED TO DETERMINE THE CONCENTRATIONS OF THE FOLLOWING COMPOUNDS:

1,3 DICHLOROBENZENE
1,4 DICHLOROBENZENE
1,2 DICHLOROBENZENE
HEXACHLOROETHANE
BIS(2-CHLOROETHYL)ETHER
NITROSODI-N-PROPYLAMINE
NITROBENZENE
HEXACHLOROBUTADIENE
1,2,4-TRICHLOROBENZENE
BIS(2-CHLOROETHOXY)METHANE
NAPHTHALENE
ISOPHORONE
HEXACHLOROCYCLOPENTADIENE
2-CHLORONAPHTHALENE
ACENAPHTHYLENE
ACENAPHTHENE
2,6-DINITROTOLUENE
4-CHLOROPHENYL PHENYL ETHER
FLUORENE
2,4-DINITROTOLUENE
DIETHYL PHTHALATE

1,2-DIPHENYLHYDRAZINE
N-NITROSODIPHENYLAMINE
HEXACHLOROBENZENE
4-BROMOPHENYL PHENYL ETHER
ANTHRACENE
PHENANTHRENE
DI-N-BUTYL PHTHALATE
FLUORANTHENE
PYRENE
BIS(2-ETHYLHEXYL)PHTHALATE
DI-N-OCTYL PHTHALATE
CHRYSENE
BENZO(A)ANTHRACENE
BENZO(A)PYRENE
INDENO(1,2,3-C,D)PYRENE
DIBENZO(A,H)ANTHRACENE
BENZO(G,H,I)PERYLENE

A. TESTED CONCENTRATION RANGE: 4-100 µg/L

B. SENSITIVITY: SEE FEDERAL REGISTER

C. DETECTION LIMITS: (UGL, BY HUBAUX + VOS)

1,3-DICHLOROBENZENE	19.5
1,4-DICHLOROBENZENE	17.6
1,2-DICHLOROBENZENE	19.4
HEXACHLOROETHANE	17.1
BIS(CHLORO ETHYL)ETHER	24.8

NITROSODI-N-PROPYLAMINE	5.8
NITROBENZENE	15.0
HEXACHLOROBUTADIENE	22.0
1,2,4-TRICHLOROBENZENE	13.0
BIS(2-CHLOROETHOXY)METHANE	9.5
NAPHTHALENE	26.8
ISOPHORONE	10.3
HEXACHLOROCYCLOPENTADIENE	44.8
2-CHLORONAPHTHALENE	12.0
ACENAPHTHYLENE	14.8
ACENAPHTHENE	13.7
2,6-DINITROTOLUENE	17.5
4-CHLOROPHENYL PHENYL ETHER	16.6
FLUORENE	15.7
2,4-DINITROTOLUENE	36.7
DIETHYL PHTHALATE	24.4
1,2-DIPHENYLHYDRAZINE	15.1
N-NITROSODIPHENYLAMINE	13.5
HEXACHLOROBENZENE	18.8
4-BROMOPHENYL PHENYL ETHER	15.1
ANTHRACENE	11.8
PHENANTHRENE	7.2
DI-N-BUTYL PHTHALATE	40.3
FLUORANTHENE	9.8
PYRENE	7.5

BIS(2-ETHYLHEXYL)PHTHALATE	88.0
DI-N-OCTYL PHTHALATE	54.2
CHRYSENE	11.9
BENZO(A)ANTHRACENE	10.8
BENZO(A)PYRENE	19.8
INDENO(1,2,3-C,D)PYRENE	28.3
DIBENZO(A,H)ANTHRACENE	29.2
BENZO(G,H,I)PERYLENE	28.2

D. INTERFERENCES: SEE FEDERAL REGISTER 12/3/79 VOL. 4, NO. 233

E. ANALYSIS RATE: AFTER SAMPLE EXTRACTION AND CONCENTRATION ONE ANALYST CAN ANALYZE ONE SAMPLE PER HOUR

CHEMISTRY

C6H4CL2 1,3-DICHLOROBENZENE
CAS RN 541-73-1
MELTING PT -24C BOILING PT 172-3C

C6H4C12 1,4-DICHLOROBENZENE
CAS RN 106-46-7
MELTING PT 54-56C BOILING PT 173C

C6H4CL2 1,2-DICHLOROBENZENE
CAS RN 95-50-1
MELTING PT -17C BOILING PT 178C

C2CL6 HEXACHLOROETHANE
CAS RN 67-72-1
MELTING PT 190-195C

C4H8CL2O BIS(2-CHLOROETHYL)ETHER
CAS RN 111-44-4
MELTING PT -50C BOILING PT 178C

C6H14N2O NITROSODI-N-PROPYLAMINE
CAS RN 621-64-7
MELTING PT 52-53C BOILING PT 312C

C6H5NO2 NITROBENZENE
CAS RN 98-95-3
MELTING PT 5-6C BOILING PT 210-211C

C4CL6 HEXACHLOROBUTADIENE
CAS RN 87-68-3
MELTING PT -19C BOILING PT 210-220C

C6H3CL3 1,2,4-TRICHLOROBENZENE
CAS RN 120-82-1
MELTING PT 16C BOILING PT 214C

$C_5H_{10}Cl_2O_2$ BIS(2-CHLOROETHOXY)METHANE

C10H8 NAPHTHALENE
CAS RN 91-20-3
MELTING PT 81-83C BOILING PT 217.7C

C9H14O ISOPHORONE
CAS RN 78-59-1
MELTING PT -8OC BOILING PT 213-214C

C5CL6 HEXACHLOROCYCLOPENTADIENE
CAS RN 77-47-4
MELTING PT -10C BOILING PT 239C

C10H7Cl 2-CHLORONAPHTHALENE
CAS RN 91-58-7
MELTING PT 59.5C BOILING PT 256C

C12H8 ACENAPHTHYLENE
CAS RN 208-96-8
MELTING PT 88-91C BOILING PT 280C

C12H10 ACENAPHTHENE
CAS RN 83-32-9
MELTING PT 93-95C BOILING PT 279C

C7H6N2O4 2,6-DINITROTOLUENE
CAS RN 606-20-2
MELTING PT 64-66C

$C_{12}H_9ClO$ 4-CHLOROPHENYL PHENYL ETHER
CAS RN 7005-72-3

C13H10 FLUORENE
CAS RN 86-73-7
MELTING PT 112-115C BOILING PT 298C

C7H6N2O4 2,4-DINITROTOLUENE
CAS RN 121-14-2
MELTING PT 69.5 BOILING PT 300C

C12H14 O4 DIETHYL PHTHALATE
CAS RN 84-66-2
MELTING PT -3C BOILING PT 298-299C

C12H12N 2 1,2-DIPHENYLHYDRAZINE
CAS RN 122-66-7
MELTING PT 123-126C

C12H10N2O N-NITROSODIPHENYLAMINE
CAS RN 1689-82-3
MELTING PT 144-145C

C6Cl6 HEXACHLOROBENZENE
CAS RN 118-74-1
MELTING PT 227-229C BOILING PT 332 C

C12H9BrO 4-BROMOPHENYL PHENYL ETHER
CAS RN 101-55-3
MELTING PT 18C BOILING PT 305C

C14H10 ANTHRACENE
CAS RN 120-12-7
MELTING PT 214-217C BOILING PT 340C

C14H10 PHENANTHRENE
CAS RN 85-01-8
MELTING PT 99-101C BOILING PT 336C

C16H2204 DI-N-BUTYL PHTHALATE
CAS RN 84-74-2
MELTING PT -35C BOILING PT 340C

C16H10 FLUORANTHENE
CAS RN 206-44-0
MELTING PT 107-110C BOILING PT 384C

C16H10 PYRENE
CAS RN 129-00-0
MELTING PT 149-151C BOILING PT 404C

C24H3804 BIS(2-ETHYLHEXYL)PHTHALATE
CAS RN 117-81-7
MELTING PT -50C BOILING PT 384C

C24H3804 DI-N-OCTYL PHTHALATE
CAS RN 117-81-7
MELTING PT - 50°C BOILING PT 384°C

C18H12 CHRYSENE
CAS RN 218-01-9
MELTING PT 250-253C BOILING PT 448C

C18H12 BENZO(A)ANTHRACENE
CAS RN 56-55-3
MELTING PT 157-159C BOILING PT 437.6C

C20H12 BENZO(A)PYRENE
CAS RN 50-32-8
MELTING PT 179C

C22H12 INDENO (1,2,3,-C,D)PYRENE
CAS RN 193-39-5
MELTING PT 162.5-164C

C22H14 DIBENZO(A,H)ANTHRACENE
CAS RN 53-70-3
MELTING PT 266C BOILING PT 524C

C22H12 BENZO(G,H,I)PERYLENE
CAS RN 191-24-2
MELTING PT 277-279C BOILING PT >750OC

USE CAUTION IN HANDLING, TOXIC INHALATION AND SKIN ABSORPTION HAZARDS EXIST.

APPARATUS:

A. INSTRUMENTATION:

FINNIGAN 4000 GAS CHROMATOGRAPH/MASS SPECTROMETER WITH A 6110 DATA AQUISITION SYSTEM.

B. PARAMETERS:

COLUMN: 15 METER FUSED SILICA CAPILLARY COLUMN COATED WITH SP2100.

TEMPERATURE PROGRAM: 30°C FOR ONE MINUTE: HEAT RAPIDLY TO 50°C THEN LINEARLY TO 260°C AT 5°/MIN

HYDROGEN CARRIER GAS: 2 ML/MIN

INJECTOR: 260°C GROB-TYPE SPLITLESS BY HOT NEEDLE TECHNIQUE

TRANSFER LINE: 200°C

SEPARATOR: 200°C

MASS SPECTROMETER SCAN EI DATA OVER MASS RANGE OF 41-330 AMU EVERY 1.18 SECS.

C. HARDWARE/GLASSWARE: SEE FEDERAL REGISTER

D. CHEMICALS: SEE FEDERAL REGISTER

STANDARDS

A. CALIBRATION STANDARDS:

STOCK A: WEIGH 20 UG OF EACH BASE-NEUTRAL COMPOUND INTO A 100 ML VOLUMETRIC AND DILUTE TO VOLUME IN MEOH.

INTERNAL STANDARD STOCK: WEIGH 5 MG D_{10} ANTHRACENE INTO A 10 ML VOLUMETRIC AND DILUTE TO VOLUME IN MEOH. ADD 10 UL PER 2 ML FOR EVERY SAMPLE AND CALIBRATION STANDARD IMMEDIATELY BEFORE ANALYSIS.

	UL STOCK ADDED TO 2ML CH_2Cl_2	PPB
CAL 1	20 UL	2
CAL 2	50 UL	5
CAL 3	100 UL	10
CAL 4	200 UL	20
CAL 5	400 UL	40

B. CONTROL SPIKES: PREPARE USING STOCK A AND NATURAL WATER

		Conc. of 2 mL CH_2Cl_2 Extr. (UG/L)
QC 1	BLANK	-
QC 2	10 UL/L	1
QC 3	25 UL/L	2.5
QC 4	50 UL/L	4.0
QC 5	100 UL/L	10
QC 6	250 UL/L	20

PROCEDURE: SEE FEDERAL REGISTER 12/3/79. Vol. 44, No. 233

CALCULATIONS:

CALCULATE UGL FOR EACH BASE-NEUTRAL AND EACH SAMPLE FROM DAILY CALIBRATION DATA. USING CONTROL SPIKE DATA, PLOT UGL ADDED VERSUS UGL FOUND BY THE METHOD OF HUBAUX AND VOS USING DL TAPE SUPPLIED BY USATHAMA. CORRECT FIELD SAMPLE CONCENTRATIONS USING THE SLOPE OF THE LINEAR REGRESSION LINE.

REFERENCE:

FEDERAL REGISTER 12/3/79. VOL. 44, NO. 233.
PGS. 69540-69547.

TABLE C-1
QUALITY CONTROL SAMPLES[a]
TASK R902.35.08
BASE/NEUTRALS (µg/L)

SPIKE SAMPLE	+4	+10	+20	+40	+100	m	R	x(d)
1,3 Dichlorobenzene	-	-	6.3	20.8	55.5	.581	.995	19.5
1,4 Dichlorobenzene	-	0.6	8.1	11.2	54.5	.570	.996	17.6
1,2 Dichlorobenzene	-	0.7	8.4	24.6	57.6	.604	.995	19.4
Hexachloroethane	-	1.8	6.3	21.6	56.7	.590	.996	17.1
Bis(2-chloroethyl) ether	-	-	9.5	43.4	104.2	1.10	.992	24.8
Nitrosodi-n-propylamine	4.0	8.3	14.9	33.2	78.8	.788	.999	5.8
Nitrobenzene	2.3	4.8	11.7	33.5	93.4	.954	.997	15.0
Hexachlorobutadiene	-	-	5.3	21.0	53.6	.564	.994	22.0
1,2,4-Trichlorobenzene	-	2.1	9.6	26.3	65.6	.682	.998	13.9
Bis(2-chloroethoxy) methane	0.4	5.7	13.6	34.7	85.7	.889	.999	9.5
Naphthalene	-	-	11.2	37.4	79.9	.846	.991	26.8
Isophorone	1.3	4.3	13.3	33.4	86.6	.889	.999	10.3
Hexachlorocyclopentadiene[b]	13.3	18.0	28.2	54.5	114.9	.421	.989	44.8
2-Chloronaphthalene	-	4.1	11.9	32.1	78.5	.812	.998	12.0
Acenaphthylene	1.4	5.6	13.2	32.6	85.2	.878	.997	14.8
Acenaphthene	-	5.6	15.7	38.0	86.4	.892	.998	13.7
2,6-Dinitrotoluene	-	-	13.2	26.7	73.0	.758	.996	17.5
4-Chlorophenyl phenyl ether	-	2.7	12.5	35.5	82.9	.865	.996	16.6
Fluorene	-	3.4	12.8	36.0	84.1	.875	.997	15.7
2,4-Dinitrotoluene	-	-	-	27.8	74.2	.788	.983	36.7
Diethyl phthalate	0.1	3.3	2.6	7.4	18.7	.187	.992	24.4
1,2-Diphenylhydrazine	3.2	6.2	13.5	35.1	101.5	1.03	.997	15.1
N-Nitrosodiphenylamine	-	9.1	16.4	39.6	89.4	.914	.998	13.5
Hexachlorobenzene	0.2	5.0	12.3	32.2	68.4	.707	.996	18.8
4-Bromophenyl phenyl ether	0.2	5.1	13.6	36.3	81.8	.846	.997	15.1

[a] Spike levels are 0, 12, 30, 60, 120 ppb.

[b] Field sample from uncontaminated background well bordering a U.S. Army installation site.

m = slope

QUALITY CONTROL SAMPLES (Cont'd.)

BASE/NEUTRALS (µg/L)

SPIKE SAMPLE	+4	+10	+20	+40	+100	m	R	x(d)
Anthracene[c]	-	2.6	6.0	18.3	37.4	.779	.993	11.8
Phenanthrene [c]	-	2.9	6.9	18.6	42.3	.874	.997	7.2
Di-n-butyl phthalate	0.5	6.8	3.3	10.3	29.1	.284	.980	40.3
Fluoranthene [c]	0.1	3.2	6.7	18.2	39.0	.805	.996	9.8
Pyrene[c]	-	3.0	6.6	17.4	38.9	.802	.997	7.5
Bis(2-ethylhexyl) phthalate[c]	17.1	21.2	26.6	46.2	62.2	.545	.927	88.0
Di-n-octyl phthalate [c]	7.7	10.3	13.4	22.2	27.8	.485	.905	54.2
Chrysene [c]	0.1	3.0	6.8	17.2	34.2	.706	.993	11.9
Benzo(a)anthracene [c]	-	4.7	7.1	15.8	32.8	.665	.994	10.8
Benzo(a)pyrene [c]	2.2	4.0	6.6	16.6	27.6	.552	.981	19.8
Indeno (1,2,3-c,d) pyrene[c]	-	5.5	8.0	15.4	24.3	.486	.963	28.3
Dibenzo (a,h) Anthracene [c]	-	6.2	8.2	16.2	25.4	.506	.961	29.2
Benzo (g,h,i) perylene[c]	-	5.0	7.2	15.4	23.8	.481	.963	28.2

[c] Spike levels are 0, 2, 5, 10, 20, 50 ppb.

m = slope

TABLE C-2
SUMMARY OF QUALITY CONTROL DATA

PRIORITY POLLUTANT CHEMICAL ANALYSIS
BASE/NEUTRALS

	Method Ref. Std.[a]					Raw Waste. Spike[b]		
SAMPLE NUMBER	$\bar{P}$	Sp	%Sp			$\bar{P}$	Sp	%Sp
1,3 Dichlorobenzene 1,4 Dichlorobenzene 1,2 Dichlorobenzene	68	26	38			60[c]	16	27
Hexachloroethane	62	25	41			66	38	58
Bis(chloromethyl)ether	-					-		
Bis(2-chloroethyl) ether	70	22	32			65	18	28
Bis(2-chloroisopropyl) ether	d					d		
N-Nitrosodimethylamine	12	10	85			17	11	66
Nitrosodi-n-propylamine	40	47	116			77	17	22
Nitrobenzene	70	12	16			82	11	13
Hexachlorobutadiene	63	25	40			55	11	20
1,2,4-Trichlorobenzene	73	27	36			69	9	14
2-Chloroethyl vinyl ether	-					-		
Bis(2-chloroethoxy) methane	74	21	29			52	19	37
Naphthalene	74	8	10			81	5	6
Isophorone	74	19	26			69	20	29
Hexachlorocyclopentadiene	43	31	73			6	5	87
2-Chloronaphthalene	78	18	23			77	11	14
Acenaphthylene	77	14	18			110	35	32
Acenaphthene	82	13	16			81	10	12
Dimethyl phthalate	16	15	93			18	14	77
2,6-Dinitrotoluene	76	12	16			82	14	18
4-Chlorophenyl phenyl ether	88	35	40			79	15	19
Fluorene	87	21	24			80	11	13
2,4-Dinitrotoluene	77	10	14			73	14	19
Diethyl phthalate	40	30	74			39	15	26
1,2-Diphenylhydrazine	71	7	10			73	12	17
N-Nitrosodiphenylamine	76	20	27			85	26	31
Hexachlorobenzene	74	32	43			74	27	37
4-Bromophenyl phenyl ether	84	14	17			90	23	26

a = Based on 4 data points
b = Based on 3 data points
c = Based on 2 data points
d = Standard not available

See text, page 5, for explanation of statistics.

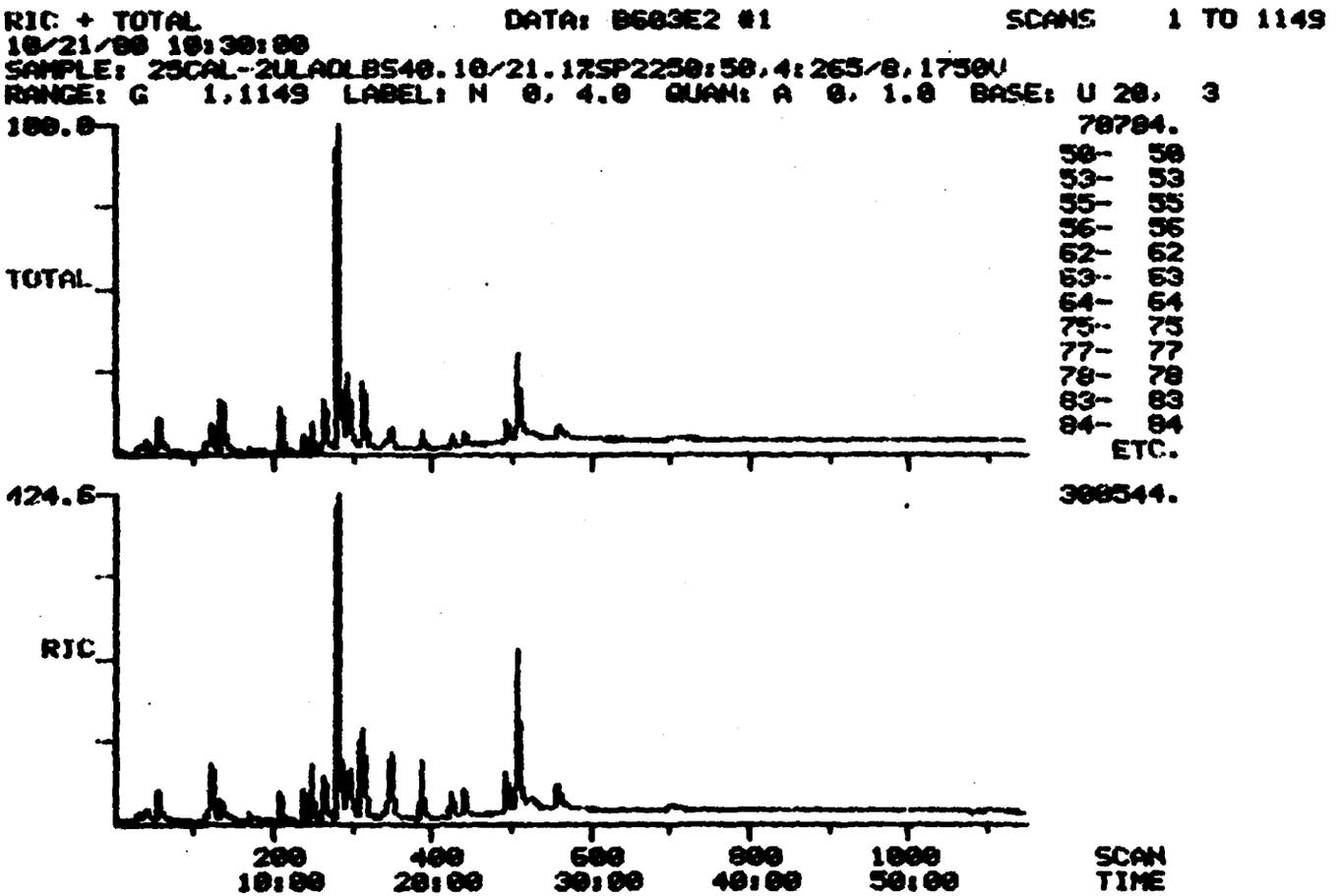
SUMMARY OF QUALITY CONTROL DATA

(con't)

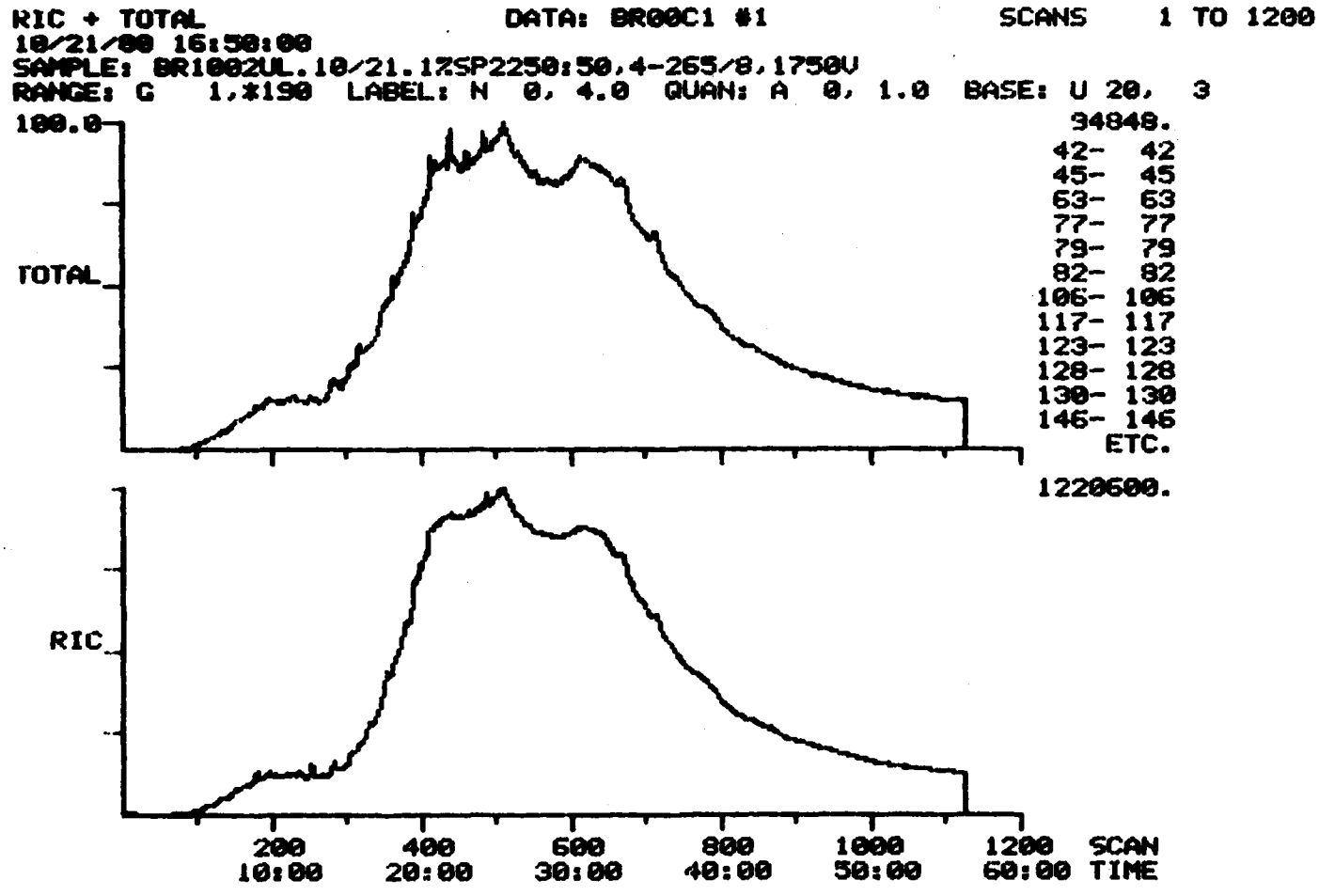
PRIORITY POLLUTANT CHEMICAL ANALYSIS
BASE/NEUTRALS

	Method Ref. Std.[a]					Raw Waste. Spike[b]		
SAMPLE NUMBER	$\bar{P}$	Sp	%Sp			$\bar{P}$	Sp	%Sp
Anthracene / Phenanthrene	84	7	9			85	19	22
Di-n-butyl phthalate	53	37	69			66	20	31
Fluoranthene	74	17	24			84	24	29
Pyrene	74	16	22			108	48	44
Benzidine	-					-		
Butyl benzyl phthalate	34	23	68			29	14	50
Bis(2-ethylhexyl) phthalate / Di-n-octyl phthalate	12	8	62			22	16	72
Chrysene / Benzo(a)anthracene	50	39	79			59	20	34
3,3'-Dichlorobenzidine	40	52	128			-		
Benzo(b)fluoranthene / Benzo(k)fluoranthene	d							
Benzo(a)pyrene	42	46	110			45	16	36
Indeno (1,2,3-c,d) pyrene	34	47	139			-		
Dibenzo (a,h) Anthracene	22	44	200			-		
Benzo (g,h,i) perylene	35	51	146			11	18	173

a = Based on 4 data points
b = Based on 3 data points
c = Based on 2 data points
d = Standard not available



Base - Neutral Calibration Mix



BR 100 Base-Neutral Fraction

APPENDIX D

Method for the Analyses of Pesticides in Water

Priority Pollutant Pesticide/PCB Data

ANALYSIS OF PESTICIDES IN WATER

APPLICATION:

METHOD USED TO DETERMINE THE CONCENTRATIONS OF THE FOLLOWING COMPOUNDS IN WATER SAMPLES:

ALPHA-BHC	ABHC
GAMMA-BHC	GBHC
HEPTACHLOR	HPCL
BETA-BHC	BBHC
DELTA-BHC	DBHC
ALDRIN	ALDRN
HEPTACHLOR EPOXIDE	HPCLE
ENDOSULFAN I	ENDOI
DDE	DDE
DIELDRIN	DLDRN
ENDRIN	ENDRN
DDD	DDD
ENDOSULFAN II	ENDOII
DDT	DDT
PCB 1254	1254
ENDOSULFAN SULFATE	ENDOS

A. TESTED CONCENTRATION RANGE: 1-10 UGL

B. SENSITIVITY:

GC/ECD TUNE REQUIREMENTS: RESPONSE TO 0.05NG ALDRIN MUST BE AT LEAST 50% FULL SCALE

C. DETECTION LIMIT: (UGL BASED ON HUBAUX + VOS)

ALPHA-BHC	1.3
GAMMA-BHC	0.4
HEPTACHLOR	0.6
BETA-BHC	0.8
DELTA-BHC	0.3
ALDRIN	0.2
HEPTACHLOR EPOXIDE	0.9
ENDOSULFAN I	0.8
DDE	0.4
DIELDRIN	0.4
ENDRIN	0.7
DDD	0.5
ENDOSULFAN II	
DDT	0.7
PCB-1254	0.7
ENDOSULFAN SULFATE	2.5

D. INTERFERENCES: NONE ENCOUNTERED

E. ANALYSIS RATE: AFTER SAMPLE EXTRACTION AND PREPARATION, AND INSTRUMENT CALIBRATION, ONE ANALYST CAN ANALYZE 8 EXTRACTS IN AN 8-HOUR DAY.

CHEMISTRY

ALPHA-BHC $C_6 H_6 CL_6$
CAS RN 319-84-6
BP 288°C MP 158°C

GAMMA-BHC $C_6 H_6 CL_6$
CAS RN 58-89-9
BP 323°C MP 113°C

HEPTACHLOR $C_{10} H_5 CL_7$
CAS RN 76-44-8
MP 96°C

BETA-BHC $C_6 H_6 CL_6$
CAS RN 319-85-7
MP 312°C

DELTA-BHC $C_6 H_6 CL_6$
CAS RN 319-86-8
MP 142°C

ALDRIN $C_{12} H_8 CL_6$
CAS RN 309-00-2
MP 104°C

HEPTACHLOR EPOXIDE $C_{11} H_5 OCL_7$
CAS RN 1024-57-3

ENDOSULFAN I $C_9 H_6 CL_6 O_3 S$
CAS RN 959-98-8
MP 108°C

DDE $C_{14} H_8 CL_4$
CAS RN 72-55-9
MP 88°C

DIELDRIN $C_{12} H_8 CL_6 O$
CAS RN 60-57-1
MP 176°C

ENDRIN $C_{12}H_8CL_6O$
CAS RN 72-20-8
MP 245°C

DDD $C_{14}H_{10}CL_4$
CAS RN 72-54-8
MP 110°C

ENDOSULFAN II $C_9H_6CL_6O_3S$
CAS RN 33213-65-9
MP 208°C

DDT $C_{14}H_9CL_5$
CAS RN 50-29-3
MP 107°C

PCB 1254 $C_{12}CL_5H_5$
CAS RN 11097-69-1
MP

ENDOSULFAN SULFATE $C_9H_6CL_6O_4S$
CAS RN 1031-07-8

USE CAUTION IN HANDLING, POTENTIAL TOXIC INHALATION AND SKIN ABSORPTION HAZARDS EXIST.

APPARATUS

A. INSTRUMENTATION: HEWLETT PACKARD GAS CHROMATOGRAPH 5840A EQUIPPED WITH AN ELECTRON CAPTURE DETECTOR.

B. PARAMETERS:

COLUMN: 1.5% SP 2250, 1.95% SP 2401, 4MM ID, 6" SPAN, 180 CM.
CARRIER GAS: 5% METHANE, 95% ARGON
TEMPERATURE: 205°C (WHILE RUNNING), 225°C (BAKING)
RUN TIME: 30 MINUTES
INJECTION PORT TEMPERATURE: 250°C
ECD TEMPERATURE: 300°C
CHART SPEED: 0.50
ZERO: 10.0
ATTENUATION: 2↑8
SLOPE SENSITIVITY: 0.15
AREA REJECTION: 1,000,000
FLOW: 75 ML
AREA REJECTION AT 1.00 MINUTE: 5000
ATTENUATION AT 18 MINUTES: 2↑7
OPTION 3: 1,2
INJECTION VOLUME: 2 UL

C. HARDWARE/GLASSWARE:

KUDERNA-DANISH GLASSWARE INCLUDING THREE BALL MACRO SNYDER COLUMNS: 500 ML EVAPORATOR FLASK: 10 ML GRADUATED RECEIVER AMPULE: AMPULE STOPPER.

SEPARATORY FUNNELS - 125, 1000 AND 2000 ML, WITH TEFLON STOPCOCKS.
CHROMAFLEX COLUMN, 400 MM LONG X 19 MM (KONTES K-420540-9011)
CENTRIFUGE BOTTLES - 250 ML
CENTRIFUGE
GRADUATED CYLINDERS - 100, 250, 500, 1000 ML
BEAKERS - 100, 400, 600 ML
SYRINGES - 10 - 500 UL
AMBER GLASS BOTTLES WITH TEFLON LINED CAPS, 500 ML AND 1-L
GLASS WOOL - METHYLENE CHLORIDE EXTRACTED
FLORISIL - PR GRADE 60-100 MESH

D. CHEMICALS:

HEXANE, FISHER BRAND, PESTICIDE GRADE
METHYLENE CHLORIDE, FISHER BRAND, PESTICIDE GRADE
METHANOL, FISHER BRAND, PESTICIDE GRADE
ETHYL ETHER, FISHER BRAND, PESTICIDE GRADE
PETROLEUM ETHER, FISHER BRAND, PESTICIDE GRADE
SODIUM SULFATE, GRANULAR, ANHYDROUS, CONDITIONED FOR TWO HOURS AT 550°C

PESTICIDE STANDARDS OBTAINED FROM EPA HEALTH EFFECTS RESEARCH LABORATORY, ENVIRONMENTAL TOXICOLOGY DIVISION, RESEARCH TRIANGLE PARK, NORTH CAROLINA.

TETRACHLOROTETRAHYDRONAPHTHALENE (TCTHN, INTERNAL STANDARD), EASTMAN CHEM. CO.

A. CALIBRATION STANDARDS:

STOCK A: WEIGH 25 MG OF EACH PESTICIDE INTO A SINGLE 25 ML VOLUMETRIC AND DILUTE TO VOLUME WITH MEOH TO MAKE A 1 MG/ML SOLUTION

DILUTION A: TAKE 1 ML STOCK A AND DILUTE TO 10 ML WITH MEOH TO MAKE A 0.1 MG/ML SOLUTION

INTERNAL STANDARD STOCK: WEIGH 20 MG TCTHN INTO 100 ML VOLUMETRIC AND DILUTE TO VOLUME WITH MEOH TO MAKE A 0.2 MG/ML SOLUTION. ADD 10 UL OF INTERNAL STANDARD TO EVERY 10 ML CALIBRATION SOLUTION AND SAMPLE IMMEDIATELY BEFORE ANALYSIS (10 UL/10 ML)

CALIBRATION STANDARDS:

		UG/ML
CAL 1	1 UL DIL. A/10ML HEXANE	0.01
CAL 2	2.5 UL DIL.A/10ML HEXANE	0.025
CAL 3	5.0 UL DIL.A/10ML HEXANE	0.05
CAL 4	7.5 UL DIL.A/10ML HEXANE	0.075
CAL 5	10 UL DIL.A/10ML HEXANE	0.1

B. CONTROL SPIKES: PREPARE USING DILUTION A STOCK, LISTED ABOVE, AND NATURAL WATER:

	UG/ML	PPB*
QC 1-BLANK	-	-
QC 2-10 UL DIL.A/L	0.01	1
QC 3-25 UL DIL.A/L	0.025	2.5
QC 4-50 UL DIL.A/L	0.05	5
QC 5-75 UL DIL.A/L	0.075	7.5
QC 6-100 UL DIL.A/L	0.1	10

*BASED on 100 ML FINAL VOLUME

PROCEDURE:

SEE REF. 1 FOR LIQUID-LIQUID EXTRACTION, FOR KUDERNA-DANISH CONCENTRATION, FOR FLORISIL PREPARATION AND SAMPLE CLEANUP, AND FOR SULFUR REMOVAL PROCEDURES.

CALCULATIONS:

CALCULATE UGL FOR EACH PESTICIDE AND EACH SAMPLE FROM DAILY CALIBRATION DATA. USING CONTROL SPIKE DATA, PLOT UGL ADDED VERSUS UGL FOUND BY THE METHOD OF HUBAUX AND VOS, USING DL TAPE SUPPLIED BY USATHAMA. CORRECT FIELD SAMPLE CONCENTRATIONS USING THIS LINE AND LINEAR REGRESSION ANALYSIS.

REFERENCE

1. U.S. EPA EVALUATION OF PROTOCOLS FOR PESTICIDES AND PCB'S IN RAW WASTEWATER. EPA-600/2-79-166. PP. 87-99.

TABLE D-1

Quality Control Samples
Task R902.35.08
Pesticides
(µg/L)

Spike Level	+1.0	+2.5	+5.0	+7.5	+10	m	R	x(d)
alpha-BHC	1.39	2.51	4.70	6.77	8.69	.852	.998	1.3
gamma-BHC	0.89	2.14	4.43	6.63	8.67	.873	1.00	0.4
Heptachlor	0.79	2.04	4.24	6.25	8.12	.821	1.00	0.6
beta-BHC	0.77	2.18	4.60	6.74	8.67	.883	.999	0.8
delta-BHC	1.00	2.19	4.43	6.55	8.69	.865	1.00	0.3
Aldrin	0.86	2.07	4.10	6.12	8.25	.820	1.00	0.2
Heptachlor epoxide	0.66	2.13	4.41	6.52	8.38	.856	.999	0.9
Endosulfan I.	0.67	2.09	4.39	6.43	8.33	.850	.999	0.8
DDE	0.83	2.12	4.59	6.92	9.14	.925	1.00	0.4
Dieldrin	.96	2.38	4.97	7.28	9.68	.971	1.00	0.4
Endrin	0.65	2.07	4.25	6.30	8.22	.836	1.00	0.7
DDD } Endosulfan II	0.68	2.03	4.27	6.38	8.42	.854	1.00	0.5
DDT	0.59	2.27	4.67	7.10	9.49	.966	1.00	0.7
PCB-1254	0.86	2.01	4.40	6.56	9.06	.903	1.00	0.7
Endosulfan sulfate	-	2.55	6.85	9.31	12.2	1.30	.993	2.5

m = slope

R = correlation coefficient

x(d)= detection limit calculated by the method of Hubaux and Vos

TABLE D-2

EPA QUALITY CONTROL DATA SUMMARY

Pesticides

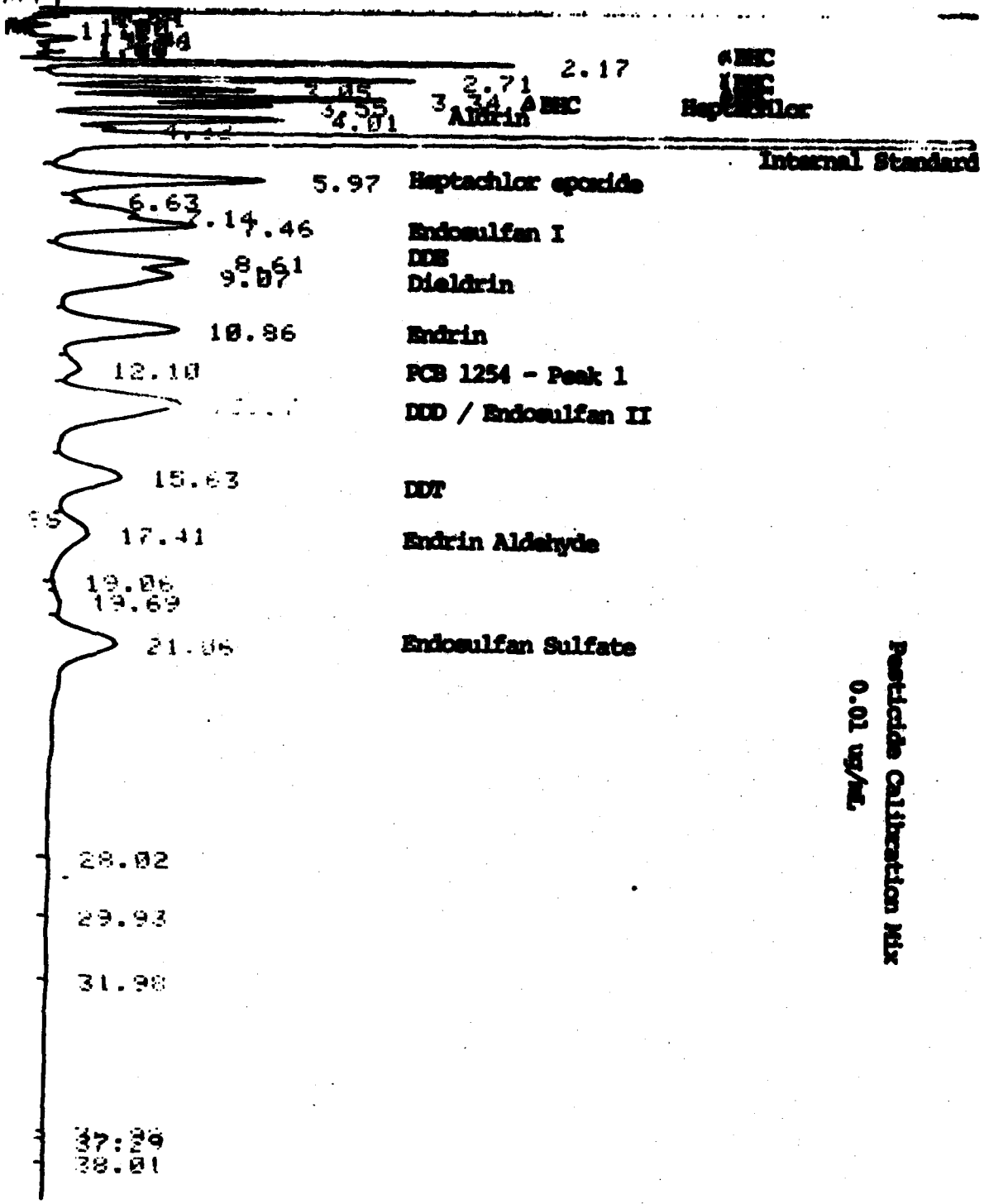
	Method Ref. Std.[a]					Raw Waste. Spike[b]		
SAMPLE NUMBER	$\bar{P}$	Sp	ZSp			P	Sp	ZSp
alpha-BHC	109	35	32			92	4	4
gamma-BHC	94	2	2			91	4	4
Heptachlor	91	1	1			85	4	4
beta-BHC	94	3	3			88	6	7
delta-BHC	93	4	4			91	4	5
Aldrin	91	4	4			83	4	4
Heptachlor epoxide	92	2	2			85	7	8
Endosulfan I.	96	3	4			95	14	14
DDE	100	8	8			91	17	19
Dieldrin	89	8	9			87	20	24
Endrin	88	2	2			93	8	8
DDD	90	6	6			88	16	18
Endosulfan II								
DDT	95	4	4			85	10	11
PCB-1254	84	7	8			74	7	9

a = Based on 4 data points

b = Based on 3 data points

See text, page 5, for explanation of statistics.

NO METHOD AREA %
START



Pesticide Calibration Mix
0.01 ug/mL

Arochlor 1016

Aroclor 1242

HR 100 6% FLORISIL FRACTION

ER 100 15% FLORISIL FRACTION

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